

REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION AD-A217 028 Final Report			1b. RESTRICTIVE MARKINGS						
6a. NAME OF PERFORMING ORGANIZATION The University of Texas at Arlington			6b. OFFICE SYMBOL (If applicable)		7a. NAME OF MONITORING ORGANIZATION Office of Naval Research				
6c. ADDRESS (City, State, and ZIP Code) Center for Advanced Polymer Research Department of Chemistry, Box 19065, University of Texas at Arlington, Arlington, TX 76019			7b. ADDRESS (City, State, and ZIP Code) 800 North Quincy Street Arlington, Virginia 22217						
8a. NAME OF FUNDING/SPONSORING ORGANIZATION Defense Advanced Research Projects Agency		8b. OFFICE SYMBOL (If applicable) DARPA		9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER N00014-86-K-0769					
8c. ADDRESS (City, State, and ZIP Code) 1410 Wilson Boulevard Arlington, Virginia 22209			10. SOURCE OF FUNDING NUMBERS		PROGRAM ELEMENT NO.		PROJECT NO.	TASK NO.	WORK UNIT ACCESSION NO.
11. TITLE (Include Security Classification) Electronic and Ionic Transport in Polymers									
12. PERSONAL AUTHOR(S) Martin Pomerantz, John R. Reynolds, Krishnan Rajeshwar and Dennis S. Marynick									
13a. TYPE OF REPORT Final Report			13b. TIME COVERED FROM 9/15/86 TO 9/30/89		14. DATE OF REPORT (Year, Month, Day) 1989, December 29			15. PAGE COUNT 10	
16. SUPPLEMENTARY NOTATION									
17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)						
FIELD	GROUP	SUB-GROUP	Electroactive polymers; Polythiophenes; Polypyrroles; Ion transport; Molecular composites; Molecular orbital calculat- ions; Organometallic polymers; Liquid crystalline polymers.						
19. ABSTRACT (Continue on reverse if necessary and identify by block number) We have prepared and studied a variety of electroactive polymers including self-doped conducting polypyrroles, high molecular weight poly(3-alkylthiophenes), new polymers containing both various aryl groups along the main chain and solubilizing substituents, electroactive polymers containing main chain transition metal complexes, fluoropolymers for high permittivity materials, liquid crystalline polymers and polymers containing PN linkages in the main chain. We have also prepared films and/or fibers of these materials both pure and as blends and molecular composites (using polyelectrolytes). We have used spectroscopy, electrochemistry and microgravimetry to study ion transport and redox switching in these systems and have developed <i>in situ</i> techniques to monitor and quantify this ion transport. We have used theoretical calculations (PRDDO and <i>ab initio</i>) to study HOMO-LUMO gaps, spin densities, structures and conformations in monomers and oligomers and have used these methods and Extended Hückel calculations to estimate band gaps and to help understand substituent effects in these polymers.									
20. DISTRIBUTION / AVAILABILITY OF ABSTRACT <input checked="" type="checkbox"/> UNCLASSIFIED/UNLIMITED <input checked="" type="checkbox"/> SAME AS RPT. <input type="checkbox"/> DTIC USERS					21. ABSTRACT SECURITY CLASSIFICATION Unclassified				
22a. NAME OF RESPONSIBLE INDIVIDUAL Dr. JoAnn Milliken					22b. TELEPHONE (Include Area Code) (202) 696-4410			22c. OFFICE SYMBOL	

DEFENSE ADVANCED RESEARCH PROJECTS AGENCY/OFFICE OF NAVAL RESEARCH
FINAL REPORT

for

Contract N00014-86-K-0769

R&T Code a400008df

Electronic and Ionic Transport in Polymers

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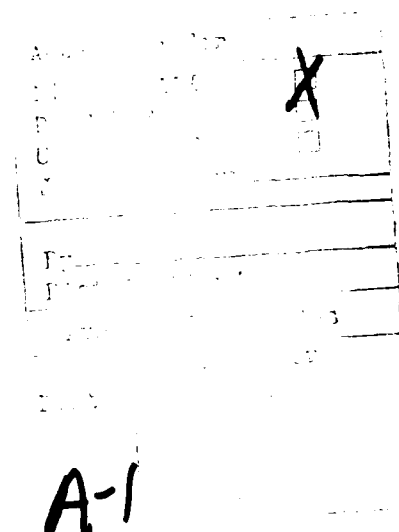
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Electronic and Ionic Transport in Polymers

This research program has involved the study of processable, high molecular weight polyheterocycles, in particular polypyrroles and poly(3-alkylthiophenes) (where alkyl = butyl – dodecyl), both neat and as blends, as fibers, films and molded forms for their electronic, electromagnetic, ion transport and mechanical properties. Polypyrrole/polyelectrolyte molecular composites have been prepared electrochemically to gain insight into the effect of carrier polymer on the material's physical, mechanical, electrical and ion transport properties. New conjugated polymers, employing various aryl groups along the main chain and solubilizing substituents, were modeled theoretically, prepared synthetically, and structurally characterized to elucidate the effect of polymer structure on the optoelectronic properties. We have also studied electroactive polymers based on main chain transition metal complexes with the goal of controlling and improving physical and electronic properties of materials for specific applications. Fluoropolymers containing a high density of attached dipoles were examined with the goal of producing materials with elevated permittivities while retaining low loss behavior. A number of polymers containing phosphorus, nitrogen and organic groups in the main chain [poly(organo- λ^5 -phosphazenes)] have been prepared and studied. We have developed novel *in situ* techniques to monitor and quantify ion transport during redox switching of conducting polymers. Spectroscopy, electrochemistry and microgravimetry have been combined to develop a detailed understanding of the dynamics of the charge and ion transport through these conductive polymers. Theoretical calculations (PRDDO and *ab initio*) have been used to calculate structures, conformations, spin densities, HOMO-LUMO gaps, and potential energy surfaces for a wide variety of heterocyclic monomers and oligomers. They were used to predict the site of electrochemical polymerization and to understand how various substituents effect the electronic and three dimensional structure of the polymers. Extended Hückel band structure calculations were performed on many of these systems to assess qualitative trends in band gaps. Liquid crystalline polyethers and polyazomethines (LCP) were synthesized and characterized both for their mesomorphic and electroactive properties. Mesogenic core units which were incorporated in these LCP's included such potential electroactive centers as extended conjugated units and transition metal chelates.

The following is a brief summary of some of the results which we have achieved and studies which have been done over the past three years.

- 1) Preparation and study of self-doped conducting polymers that have ion-specific transport and are water soluble.

- 2) Use of a quartz crystal microbalance to analyze electropolymerization and charge transport in polyheterocycles.
- 3) Synthesis of variable conductivity copolymers of pyrrole and N-substituted pyrroles; substantiated with theoretical calculations.
- 4) The conductivity of polypyrrole free standing films and anion exchanged films have been shown to be relatively insensitive to dopant anion but dependent on morphology.
- 5) Spectroelectrochemical demonstration of proton transport through a Au-supported polypyrrole film during redox switching.
- 6) Theoretical calculations of pyrrole and thiophene monomers and oligomers correlate with electropolymerization tendencies.
- 7) Model compound studies have been carried out in the areas of high permittivity, ion-conducting and liquid crystalline polymers.
- 8) Charge transport properties of electrochemically prepared poly(3-methylthiophene) thin films were shown to depend on the morphology of the film.
- 9) Soluble transition metal bisdithiolenes were observed to be electroactive with three distinct oxidation states.
- 10) Carbon felt electrodes have allowed fairly large quantities of soluble poly(3-hexylthiophene) to be prepared.
- 11) Composite polypyrrole/poly(phenyleneterephthalamide propanesulfonate) films, where the dopant is a thermally stable polyelectrolyte modified Kevlar, were shown to have good electrical conductivities of ca. $10^{-3} \text{ S cm}^{-1}$.
- 12) The diffusion coefficients for anion exchange in polypyrrole were shown to be a function of polymer morphology.
- 13) Proton transport was seen to accompany switching of polypyrrole from the conducting to the insulating state.
- 14) Redox switching of polypyrrole has been observed to modify the luminescence behavior of an interfacial probe (e.g. pyrene).
- 15) Developed of quantum mechanical methods for estimating the relative energies of the quinoid and aromatic terms of polyheterocyclic systems.
- 16) The liquid crystalline properties of aliphatic/aromatic polyazomethine ethers was shown to depend on the configuration of atoms in the isomeric mesogenic core and the LC properties of some metal chelates depends on the nature of the transition metal.
- 17) A chemical polymerization method, using FeCl_3 and oxygen, has provided high molecular weight poly(3-alkylthiophenes), (alkyl = butyl – dodecyl) in fairly large quantities.
- 18) Fibers of poly(3-decylthiophene) have been melt spun in collaboration with Hoechst-Celanese, Summit, NJ, and completely characterized.

- 19) Molecular composite membranes of polypyrrole with poly(styrenesulfonate) have been prepared, shown to have good conductivity and mechanical properties and their ion transport properties have been studied using the electrochemical quartz crystal microbalance.
- 20) Large films of polypyrrole molecular composites have been incorporated into epoxy laminate panels and their surface resistivity monitored using microwave measurements in a collaborative program with General Dynamics, Fort Worth, TX.
- 21) We have demonstrated that the fluorescence intensity of a molecule located close to a conductive polymer (e.g. polypyrrole) surface may be modulated by redox switching, and accompanying variations in the dielectric characteristics of the polymer.
- 22) We have developed *in situ* optical absorption and fluorescent probes which can monitor ion transport during redox switching of polypyrroles and polythiophenes.
- 23) We have used quantum mechanical calculations to do conformational analysis on intramolecularly hydrogen bonded copolymers containing heterocyclic and aromatic rings and to study the relative energies of aromatic and quinoid forms of polythiophene and related system. This has helped guide synthetic strategies.

The following is a listing of the journal publications and technical reports which have emanated from the contract.

Papers Published in Refereed Journals

- 1) Jang, G-W., Tsai, E. W. and Rajeshwar, K., "Electrochemically-Triggered pH Modulation at the Ruthenium Oxide/Electrolyte Interface: A Spectroelectrochemical Probe for the Proton Transport Mechanism", *J. Electrochem. Soc.*, **1987**, *134*, 2377.
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- 8) Reynolds, J.R., Sundaresan, N.S., Pomerantz, M., Basak, S. and Baker, C.K., "Self-Doped Conducting Copolymers: A Charge Transport Study of Poly{pyrrole-co-[3-(pyrrol-1-yl)propanesulfonate]}", *J. Electroanal. Chem.*, **1988**, 250, 355.
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- 10) Baker, C. K. and Reynolds, J. R., "A Quartz Microbalance Study of the Electrosynthesis of Polypyrrole", *J. Electroanal. Chem.*, **1988**, 251, 307.
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- 28) Shaffer, T. D. and Kramer, M. C., "Cyclization vs. Polymerization in Phase Transfer Catalyzed Polythioetherification", *Makromol. Chem.*, accepted for publication.
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- 34) Gieselman, M. G. and Reynolds, J. R., "Poly(*p*-phenyleneteneophthalamide propane sulfonate): A New Polyelectrolyte for Application to Conducting Molecular Composites", *Macromolecules*, submitted for publication.
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Paper Acknowledging DARPA Support for Instrumental Purchase

- 39) Marques, H. M., Scooby, D. C., Victor, M. and Brown, K. L., "Optimisation of the Preparation and Purification of Three Monocarboxylic Acid Derivatives of Vitamin B₁₂ and Their Characterisation by ¹³C NMR", *Inorg. Chim. Acta.*, **1989**, 162, 151.

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- 1) Pomerantz, M., Reynolds, J. R., Rajeshwar, K., Marynick, D. S. and Shaffer, T. D. "Electronic and Ionic Transport in Polymers", Quarterly-Technical Report No. 1, Grant No. N00014-86-K-0769 DARPA/ONR, December 12, 1986.
- 2) Pomerantz, M., Reynolds, J. R., Rajeshwar, K., Marynick, D. S. and Shaffer, T. D. "Electronic and Ionic Transport in Polymers", Quarterly-Technical Report No. 2, Grant No. N00014-86-K-0769 DARPA/ONR, March 13, 1987.
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- 5) Pomerantz, M., Reynolds, J. R., Rajeshwar, K., Marynick, D. S. and Shaffer, T. D. "Electronic and Ionic Transport in Polymers", Annual Letter Report No. 1, Grant No. N00014-86-K-0769 DARPA/ONR, September 30, 1987.
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The following are the personnel who were paid with funds from this contract.

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The following are the personnel who worked in related areas but were not paid with funds from this contract.

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